Results of Proficiency Test
SCCP in Leather/Footwear
February 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. G.A. Oosterlaken-Buijs
Correctors: ing. A.S. Noordman-de Neef & ing. M. Meijer
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CONTENTS

1 INTRODUCTION

Commercially produced Chlorinated Paraffin's (CPs) are classified according to their carbon chain length into Short Chain CPs (SCCP C_{10}-C_{13}), Medium Chain CPs (MCCP C_{14}-C_{17}) and Long Chain CPs (LCCP >C_{17}). The Chlorine content of these mixtures can vary from 30-70% depending on the application. Technical CPs are used in plasticizers and fire retardants. CPs are classified as persistent and non-biodegradable and they accumulate in the food chain. SCCPs were categorized in group 2B as possibly carcinogenic to humans from the International Agency for Research on Cancer (IARC). Since 2017, SCCP is banned under the Stockholm Convention on Persistent Organic Pollutants (annex A).

Since 2019 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of SCCP in Leather/Footwear every year. During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency test for the analysis of SCCP in Leather/Footwear.

In this interlaboratory study 50 laboratories in 18 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the SCCP in Leather/Footwear proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one leather sample of 3 grams positive on SCCP and labelled #21510.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

A batch of beige colored leather positive on SCCP was selected. The leather was grinded into small pieces and mixed thoroughly. After homogenization 70 plastic bags were filled with approximately 3 grams each and labelled #21510.
The homogeneity of the subsamples was checked by determination of SCCP in accordance with ISO18219 on 10 stratified randomly selected subsamples.

	SCCP in mg/kg
sample #21510-1	271.18
sample #21510-2	245.78
sample #21510-3	263.33
sample #21510-4	243.17
sample #21510-5	242.57
sample #21510-6	252.23
sample #21510-7	291.35
sample #21510-8	267.03
sample #21510-9	249.95
sample #21510-10	279.55

Table 1: homogeneity test results of subsamples #21510

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation in agreement with the procedure of ISO13528, Annex B2, in the next table.

	SCCP in mg/kg
r (observed)	46.6
reference method	Horwitz (n=9)
0.3 x R (reference method)	45.5

Table 2: evaluation of the repeatability of subsamples #21510

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To the participating laboratories one sample labelled #21510 was sent on January 27, 2021.

2.5 ANALYZES

The participants were requested to determine: SCCP and MCCP. It was noted in the instructions of this PT to not use less than 0.5 grams per determination to ensure the homogeneity. In the instructions was also noted not to dry or age the sample, nor determine volatile matter. It was also requested to report if the laboratory was accredited for the requested components and to report some analytical details.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In general, when no literature reproducibility is available, another target may be used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used. This should be done in order to evaluate whether the reported test results are fit-for-purpose.

The z-scores were calculated in accordance with:

$z_{(target)}$ = (test result - average of PT) / target standard deviation

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
The usual interpretation of z-scores is as follows:

	|z|	< 1	good
1 <	|z|	< 2	satisfactory
2 <	|z|	< 3	questionable
3 <	|z|		unsatisfactory

4 EVALUATION

During the execution of this proficiency test some problems occurred with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. One participant reported the test results after the extended final reporting date and four other participants did not report any test results. Not all participants were able to report all components requested.
In total 46 participants reported 82 numerical test results. Observed were 6 outlying test results, which is 7.3%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER COMPONENT

In this section the test results are discussed per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data. The abbreviations, used in these tables, are explained in appendix 4.

For the determination of SCCP and MCCP, ISO18219 is considered to be the official test method. Regretfully, ISO18219 does not contain any precision data. Therefore, the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation based on nine components (n=9).

Sample #21510

SCCP: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation (n=9).

MCCP: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation (n=9).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the estimated target reproducibility calculated with the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the estimated target reproducibility calculated with the Horwitz equation are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
SCCP	mg/kg	43	213	98	128
MCCP	mg/kg	33	1050	401	495

Table 3: reproducibilities of tests on sample #21510

Without further statistical calculations, it can be concluded that for the SCCP and MCCP determination there is a good compliance of the group of participating laboratories with the target reproducibility.

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2021 WITH PREVIOUS PTS

	February 2021	April 2020	March 2019
Number of reporting laboratories	46	53	54
Number of test results	82	102	99
Number of statistical outliers	6	7	2
Percentage of statistical outliers	7.3%	6.9%	2.0%

Table 4: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RSD) of the PTS, see next table.

Component	February 2021	April 2020	March 2019	Horwitz (n=9)
SCCP	16%	24%	31%	21-22%
MCCP	14%	22%	26%	17-21%

Table 5: development of the uncertainties (RSD) over the years

The group's performance improved compared to last year's performance.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported analytical details from the participants are listed in appendix 2.
About 85% of the reporting participants mentioned to be accredited for the determination of SCCP and/or MCCP in leather.
Prior to analysis the samples were further cut or grinded by about 20% of the reporting participants, about 80% used the samples as received.
The amount of sample intake varied between 0.5 and 2 grams, about 85% used 0.5 grams.
A vast majority of the reporting participants used n-Hexane as release solvent.
All reporting participants used an extraction time of 60 minutes and an extraction temperature of 60°C.

5 DISCUSSION

In this proficiency test for the determination of SCCP in leather it was noticed that all reporting participants were able to detect SCCP. The majority of the participants reported also the presence of MCCP.

When the results of this interlaboratory study were compared to the Leather Standard by Oeko-Tex, it was noticed that all participants would make an identical decision about the acceptability of the leather for the determined components. All reporting laboratories would have rejected the sample for all categories.

Ecolabel	baby clothes	in direct skin contact	no direct skin contact
Leather by Oeko-Tex	<50 mg/kg *)	<50 mg/kg *)	<50 mg/kg *)

Table 6: Leather Standard by OEKO-TEX

*) This concerns the sum of SCCP and MCCP

6 CONCLUSION

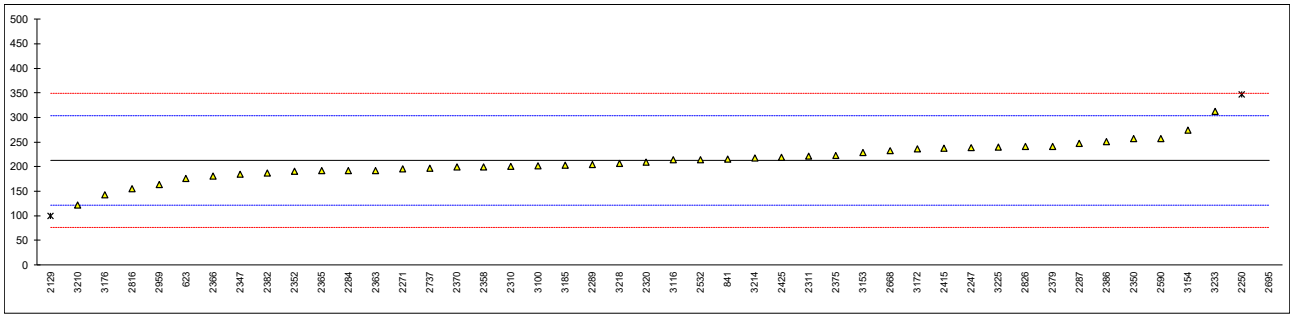
The majority of the participants is able to determine SCCP and MCCP in the leather matrix. The observed reproducibility in this proficiency test on SCCP in Leather improved for both SCCP and MCCP compared to last year.

However, each laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of SCCP on sample #21510; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	ISO18219	177.20		-0.78	
841	ISO18219	215.6		0.07	
2115		-----		-----	
2129	ISO18219	100	C,R(0.05)	-2.47	first reported 302
2247	ISO18219	239.92		0.60	
2250	ISO18219	348	C,R(0.05)	2.97	first reported 318
2271	ISO18219	196.1		-0.36	
2284	ISO18219	192.81		-0.43	
2287	ISO18219	248		0.78	
2289	ISO18219	205		-0.17	
2295		-----		-----	
2310	ISO18219	201.5		-0.24	
2311	ISO18219	221.5		0.20	
2320	ISO18219	210.1		-0.05	
2347	ISO18219	185		-0.61	
2350	ISO18219	257.3		0.98	
2352	ISO18219	191.1		-0.47	
2358	ISO18219	200.39		-0.27	
2363	ISO18219	193		-0.43	
2365	ISO18219	192.32		-0.45	
2366	ISO18219	181.4		-0.68	
2370	ISO18219	200		-0.28	
2375	ISO18219	223		0.23	
2379	ISO18219	242.1675		0.65	
2382	ISO18219	187.1		-0.56	
2386	ISO18219	252		0.87	
2390		-----		-----	
2415	ISO18219	238.08		0.56	
2425	In house	219.5		0.15	
2532	ISO18219	215		0.05	
2561		-----		-----	
2590	ISO18219	257.657		0.99	
2668	ISO18219	233		0.45	
2695	ISO18219	984.49	R(0.01)	16.95	
2737	GB/T38405	196.952		-0.34	
2816	ISO18219	155.443		-1.25	
2826	ISO18219	242.0		0.65	
2959	ISO18219	164		-1.07	
3100	ISO18219	202		-0.23	
3116	ISO18219	214.41		0.04	
3153	ISO18219	230.0		0.38	
3154	ISO18219	275	C	1.37	first reported 346.318
3172	ISO18219	236.61		0.53	
3176	ISO18219	143.40		-1.52	
3185	ISO18219	203.70		-0.20	
3210	In house	122.26		-1.98	
3214	ISO18219	218.3		0.13	
3218	ISO18219	207.79		-0.11	
3225	ISO18219	240.7		0.62	
3233	In house	313.48	C	2.21	first reported 386.74
	normality	suspect			
	n	43			
	outliers	3			
	mean (n)	212.600			
	st.dev. (n)	35.0179	RSD = 16%		
	R(calc.)	98.050			
	st.dev.(Horwitz (n=9))	45.5482			
	R(Horwitz (n=9))	127.535			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400
350
300
250
200
150
100
50
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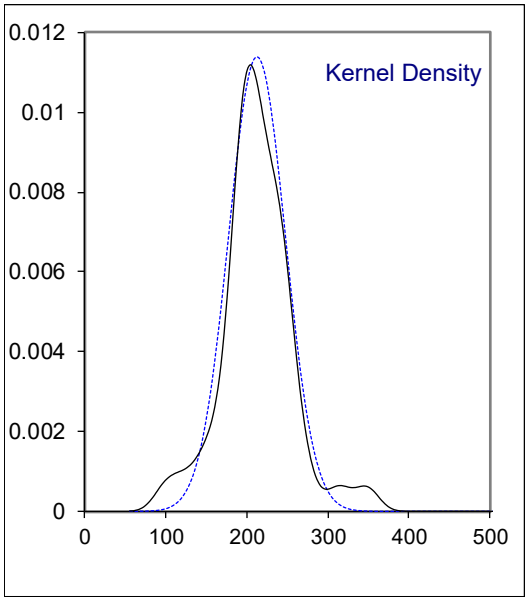
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0.008
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0.004
0.002
0
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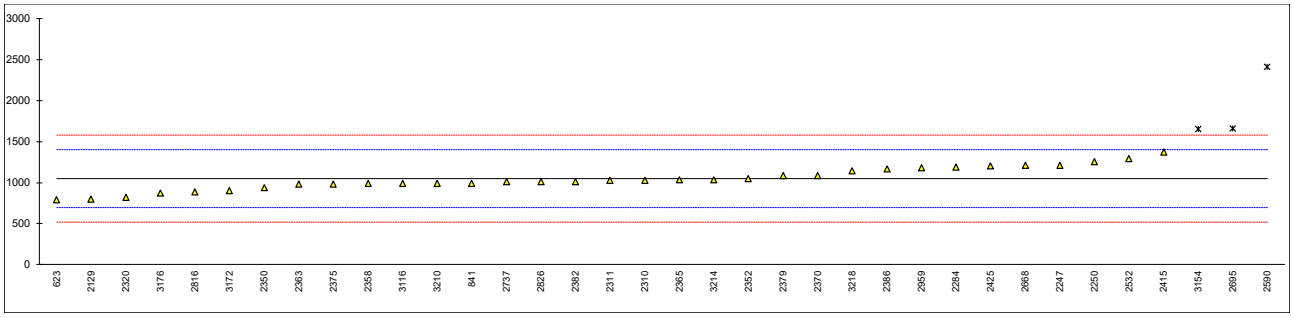
Determination of MCCP on sample #21510; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	ISO18219	792.30		-1.46	
841	ISO18219	995.9		-0.31	
2115		-----		-----	
2129	ISO18219	800	C	-1.41	first reported 1766
2247	ISO18219	1217.17		0.94	
2250	ISO18219	1262		1.20	
2271		-----		-----	
2284	ISO18219	1192.82		0.81	
2287		-----		-----	
2289		-----		-----	
2295		-----		-----	
2310	ISO18219	1031		-0.11	
2311	ISO18219	1027.9		-0.13	
2320	ISO18219	820.3		-1.30	
2347		-----		-----	
2350	ISO18219	939.39		-0.63	
2352	In house	1055.5		0.03	
2358	ISO18219	990.37		-0.34	
2363	ISO18219	988		-0.35	
2365	ISO18219	1035.75		-0.08	
2366		-----		-----	
2370	ISO18219	1090		0.22	
2375	ISO18219	988		-0.35	
2379	ISO18219	1088.8932		0.22	
2382	ISO18219	1017.8		-0.18	
2386	ISO18219	1174		0.70	
2390		-----		-----	
2415	ISO18219	1378.61		1.86	
2425	In house	1204.2		0.87	
2532	ISO18219	1296		1.39	
2561		-----		-----	
2590	ISO18219	2417.442	R(0.01)	7.73	
2668	ISO18219	1215.03		0.93	
2695	ISO18219-2Draft	1664.42	R(0.01)	3.47	
2737	GB/T38405	1014.538		-0.20	
2816	ISO18219	889.5227		-0.91	
2826	ISO18219	1015.2		-0.20	
2959	ISO18219	1182	C	0.74	first reported 1682
3100		-----		-----	
3116	ISO18219	992.38		-0.33	
3153		-----		-----	
3154	ISO18219	1655	C,R(0.01)	3.42	first reported 2186,696
3172	ISO18219	906.43		-0.81	
3176	ISO18219	873.66		-1.00	
3185		-----		-----	
3210	In house	994.62		-0.31	
3214	ISO18219	1040.1		-0.06	
3218	ISO18219	1147.76		0.55	
3225		-----		-----	
3233		-----		-----	

normality	OK
n	33
outliers	3
mean (n)	1050.216
st.dev. (n)	143.0559 RSD = 14%
R(calc.)	400.556
st.dev.(Horwitz (n=9))	176.9181
R(Horwitz (n=9))	495.371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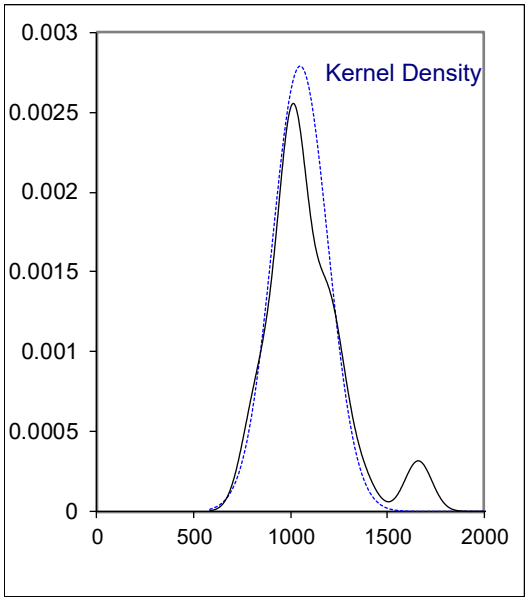
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0.002
0.0015
0.001
0.0005
0
0
500
1000
1500
2000

APPENDIX 2 Analytical details

lab	ISO17025 accredited	sample grinded or cut	intake (g)	release solvent	extraction time (min)	extraction temp (°C)	remarks
623	Yes	Further cut	0.5	hexane	60	60	
841	Yes	Used as received	0.5	toluene	60	60	
2115	---	---					
2129	---	---					
2247	Yes	Used as received	1-2	n-hexane	60.0	60.0	
2250	Yes	Used as received	0,5	hexane	60	60	
2271	Yes	Used as received	0.5	n-Hexane	60	60	
2284	Yes	Used as received	1	Hexane	60	60	
2287	No	Further cut	0.5	Hexane	60	60	
2289	Yes	Further cut	0.5	hexane	60	60	
2295	---	---					
2310	Yes	Further cut	0.5	Hexane	60	60	
2311	Yes	Further cut	0.5	Hexane	60	60	
2320	No	Used as received	0.5	Hexane	60	60	
2347	---	---	0.5	n-hexane	60	60	
2350	Yes	Used as received	0.5	Hexane	60	60	
2352	Yes	Used as received	0.5	Hexane	60	60	
2358	Yes	Used as received	0.5	n-hexane	60	60	
2363	Yes	Used as received	0.5	5mL	60	60	
2365	Yes	Used as received	0.5	Hex	60	60	
2366	Yes	Used as received	0.5	n-hexane	60	60	
2370	Yes	Used as received	0.5	Hexane	60	60	
2375	Yes	Used as received	0.5	Hexane	60	60	
2379	No	Used as received	0.5	Hexane	60	60	
2382	Yes	Used as received	0.5	toluene	60	60	
2386	Yes	Used as received	0,5	n-Hexan	60	60	Clean up with H2SO4
2390	---	---					
2415	Yes	Used as received	0.5	n-Hexane	60	60	
2425	Yes	Further cut	0.5	10ml n-hexane	1 Hour	60	
2532	Yes	Used as received	0.5001	n-Hexane	60	60	
2561	---	---					
2590	Yes	Further cut	0.5	Hexane	60	60	
2668	Yes	Further cut	0.5	n-Hexane	Sonicate for 60 min	60	
2695	Yes	Used as received	1	Hexane	60	60	
2737	Yes	Used as received	O.5	n-hexane	60	60	
2816	No	Used as received	0.5	hexane	60	60	
2826	Yes	Used as received	0.5	Toluene	60	60	
2959	No	Used as received					
3100	Yes	Used as received	0.5	n-Hexane	60±2 min	60	
3116	Yes	Used as received	1	n-Hexane	60	60	
3153	Yes	Used as received	0.5	n-hexane	60	60	
3154	Yes	---					
3172	---	---					
3176	Yes	Used as received	0,5	N-Hexane	60	60	
3185	Yes	Used as received	0.5	n-hexane	60	60	
3210	---	Used as received	1	Hexane	60	60	
3214	Yes	Used as received	0.5	Hexane	60	60	
3218	Yes	Used as received	0.5	N-hexane	60	60	
3225	Yes	Further cut	0.5	n-hexane	60	60	
3233	No	Used as received	0.95099	hexane	60	60	

APPENDIX 3

Number of participants per country

1 lab in	BANGLADESH
1 lab in	DENMARK
2 labs in	FRANCE
4 labs in	GERMANY
5 labs in	HONG KONG
5 labs in	INDIA
1 lab in	INDONESIA
4 labs in	ITALY
1 lab in	JAPAN
14 labs in	P.R. of CHINA
1 lab in	PAKISTAN
1 lab in	SOUTH KOREA
1 lab in	SRI LANKA
2 labs in	TAIWAN
1 lab in	THAILAND
3 labs in	TURKEY
1 lab in	UNITED KINGDOM
2 labs in	VIETNAM

APPENDIX 4

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

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